



INDUSTRIJA NAFTE, d.d.

SD Istraživanje i proizvodnje nafte i plina

Sektor za upravljanje i inženjering polja

Služba laboratorijskih ispitivanja IPNP

**FUEL ANALYSIS WITH SEM METHODOLOGY
(Electronic Scanner Microscope)
OF SHIP'S HFO RESIDUALS – F RME 180**

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50000360-033/13

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Date:
26 April 2013.

**FUEL ANALYSIS WITH SEM METHODOLOGY
(Electronic Scanner Microscope)
OF SHIP'S HFO RESIDUALS – F RME 180**

Responsible

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 <p>INA INDUSTRIJA NAFTE, d.d. SD Istraživanje i proizvodnje nafte i plina Sektor za upravljanje i inženjering polja Služba laboratorijskih ispitivanja IPNP</p>	<p>FUEL ANALYSIS WITH SEM METHODOLOGY (Electronic Scanner Microscope) OF SHIP'S HFO RESIDUALS – F RME 180</p>	Ref.no.: 50000360-033/13	
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		Date: 26 April 2013.	

Client: **BIMONT d.o.o.**

Senčna ulica 19
6310 Izola
Slovenia

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1. INTRODUCTION

Our Office of Geology and Geochemistry (SLI IPNP) were submitted for scanner electronic microscope analysis 3 samples of heavy ship's fuel oil, type F RME 180, manufactured by INA Rijeka Refinery.

- a) The sample labeled "0" is the residual marine heavy fuel FRME 180 before use of Homologizer TGA 3G;
- b) The sample labeled "2" is fuel emulsion after processing with Homologizer TRGA 3G, and
- c) The sample labeled "5" is the fuel emulsion after processing with Homologizer TRGA 3G and with 3% of added water to the amount of 32 liters of heavy fuel oil F RME 180.

It was necessary to establish a form of particles of Al and Si prior to processing (pattern "0") and after treatment with Homologizer TRGA 3G (samples "2" and "5").

2. TESTING METHODS

Samples of HFO are delivered into three (3) bottles, labeled as: "0", "2" and "5" (Photo 1).

In order to obtain just dry residue required for SEM analysis, HFO was necessary to be filtered first. This was done in the laboratory of geochemistry.

About the content of individual samples we received report from the client (Bimont Ltd.) as quoted:

- 0 - initial - untested sample of HFO,
- 2 - tested / analyzed sample of HFO,
- 5 – HFO sample with 3% added water.

The aim of filtering was to separate particles in HFO (defined as Alumina-Silicate material), and examination with electronic microscope shall define the shape and proportion of these particles in the submitted samples of HFO.

HFO Samples were filtered using filtration process with hot chloroform, using Büchner hopper on the nitrocellulose filter - Millipore, fineness 0.45 um. Particles remaining on the filter paper were rinsed several times with hot chloroform. Filter and particles on the filter were dried and weighed (see Table 1).



Photo 1. HFO samples

Table 1.

**Separation of sediments from HFO – hot filtration of fuel
with chloroform on a nitrocellulose filter 0,45µm**

Ref. Number of Sample	Ref. No. Of Laboratory	Sample of HFO (ml)	Mass of HFO sample (g)	Mass of filter paper (g)	Mass of fil. paper + particles on paper (g)	Mass of particles on Filter paper (g)	Mass of particles (mg on kg of HFO)
0	GK12-260	150	244,92	0,1072	0,1096	0,0024	9,8 mg/kg
2	GK12-261	150	244,93	0,0981	0,1000	0,0019	7,7 mg/kg
5	GK12-262	150	245,63	0,1042	0,1057	0,0015	6,1 mg/kg

After the filtration process 3 dry residual on the filter paper were obtained (Photo 2), those are analyzed with electronic microscope. Samples were steam processed with gold, and then analyzed using scanning electronic microscope JEOL JSM-6510 LV. Several micro photography were taken.

The EDX analysis is maiden (identification of peaks of the energy spectrum of X-radiation) for individual particles using Oxford INCA X-act.

Unmarked peaks on the graph are due to steam processing with gold (Au), and the filter paper (C and F) are omitted for reasons not to be included in the quantitative chemical analysis of particles.

On the electronic microscope is not possible to determine the total amount of only requested particles.

There were taken 22 micrographs (Photo from 3 to 24), and accompanied by the 8 pictures of EDX spectra and 8 Tables (Tables from 2 to 9).

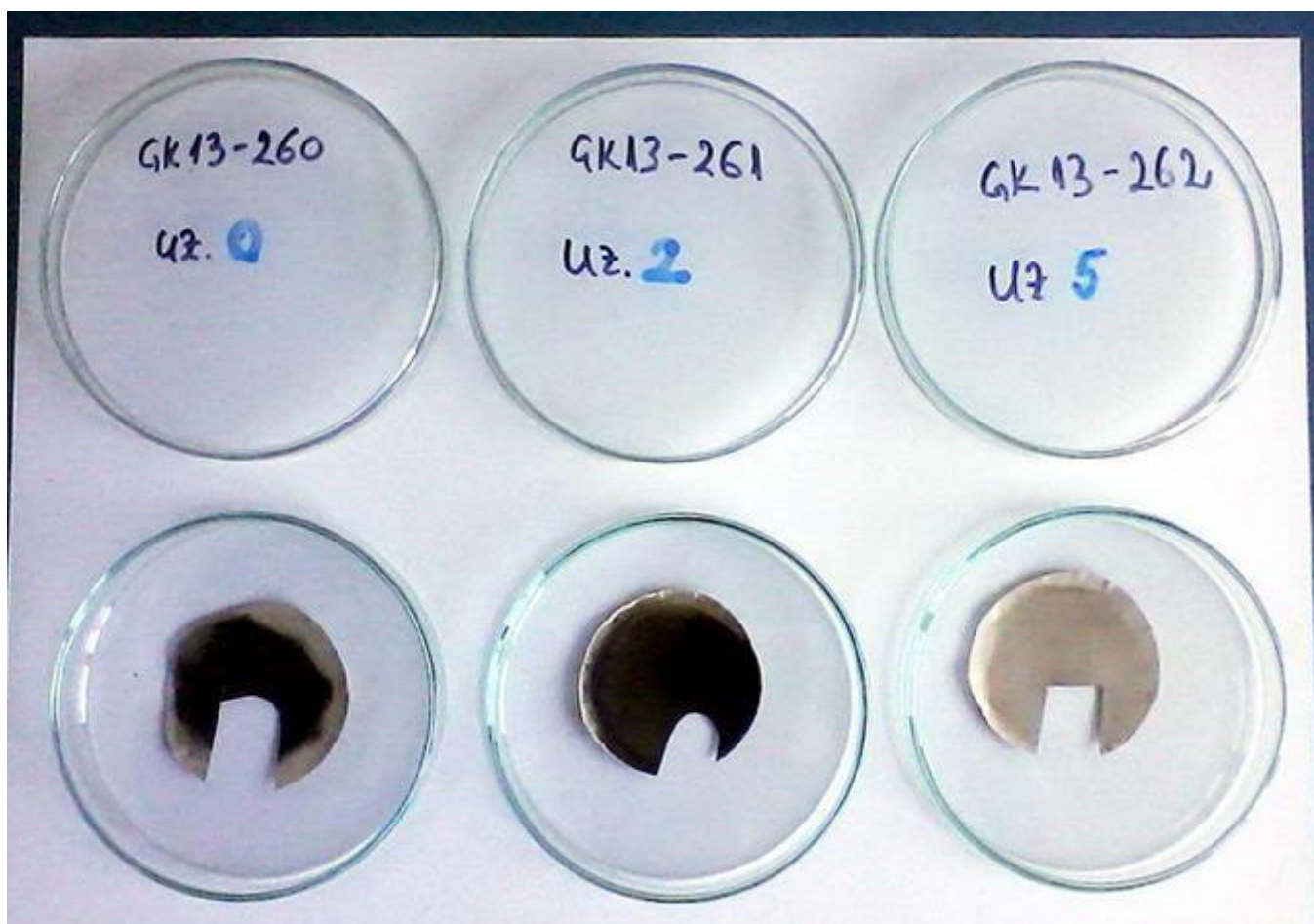


Photo 2. Samples of sediments from HFO on the filter paper

3. TEST RESULTS

Samples of "0" and "2" are quite similar, and the sample No. "5" is in macroscopic way different (lighter sediment). The sample "0" has a lot of residue on the filter paper (impurities) and sample "2" has a little less, but in the sample "5" it is almost nonexistent. Visible is only clean filter paper, which contains some particles. For comparison, for all samples were taken photo with increasing to the x500.

In all three samples were found different chemical particles. In most are present Al, Si and O, while in some particles are still present other elements, in various combinations and various ratios of elements (Mg, Fe, K, Na, Ca, S, Cu, Zn).

In all samples, the particles are of similar appearance, regardless of the composition. They are look-like scaly (layered slices with irregular edges).

In the samples "0" and "2" the particle size are about 5 to 10 μm , rarely up to 20 microns. In these were found several sediments (sizes from 10 to 30 μm) looks as fiber (thin sticks). They are composed of Si, Mg, O and little Fe, and the relationship of these elements in them varies.

Sample "5" is different from the others. The volume has much smaller particles (their size is about 1 to 5 μm).

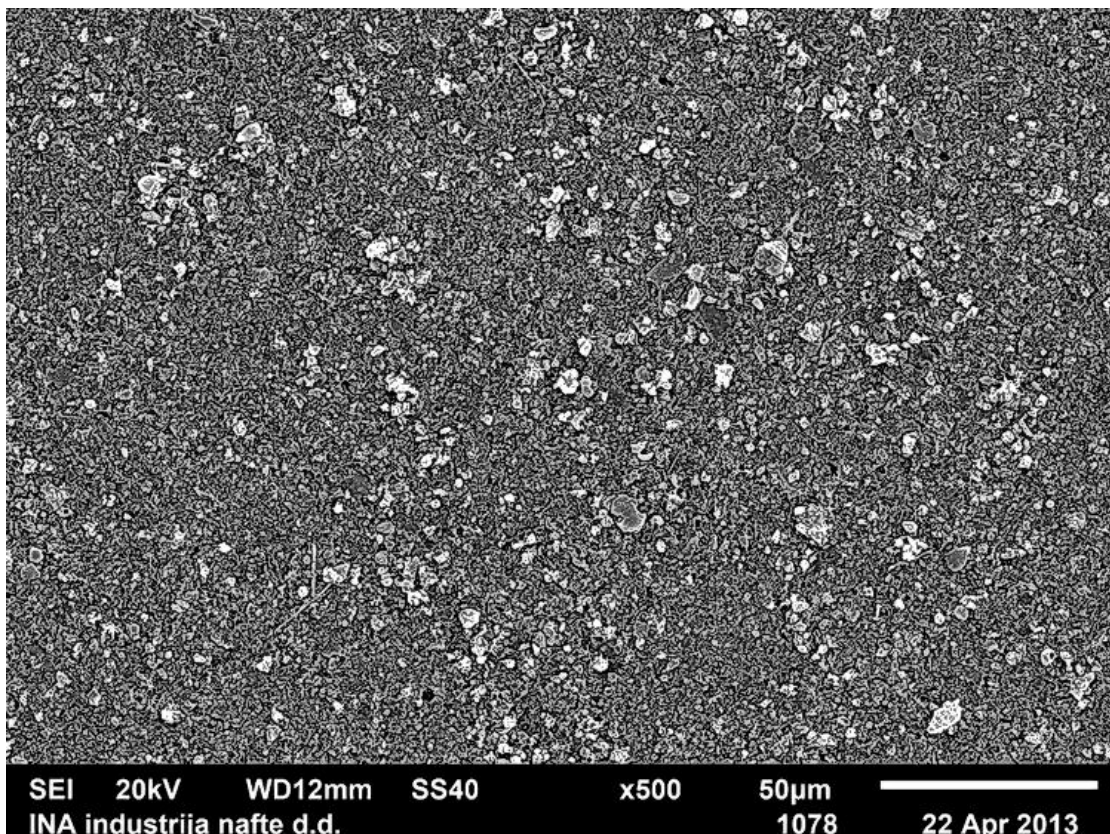


Photo 3. Sample „0“ micro photo, increasing 500x

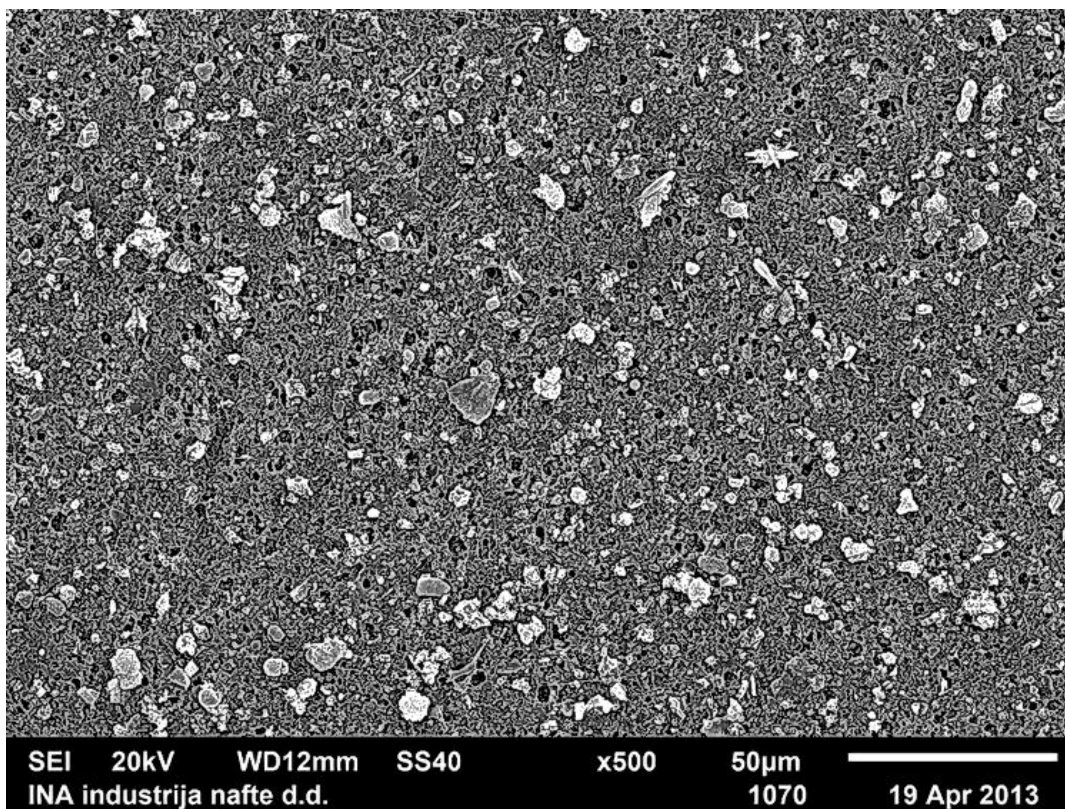


Photo 4. Sample „2“ micro photo, increasing 500x

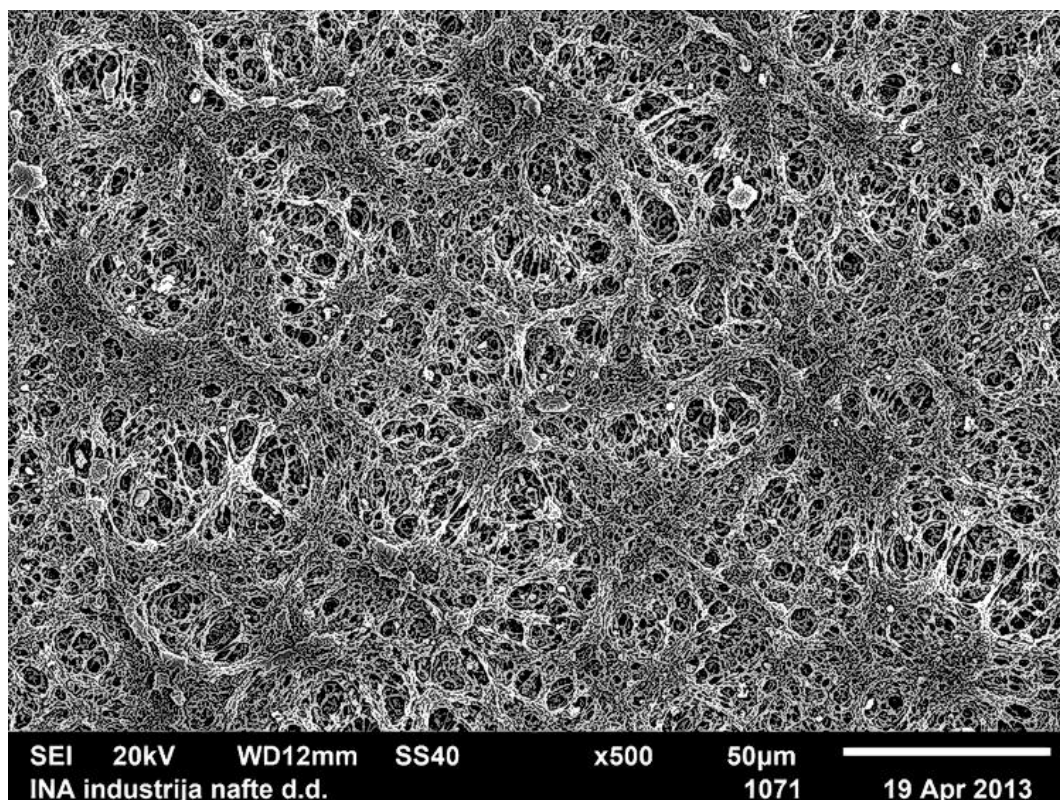


Photo 5. Sample „5“ micro photo, increasing 500x

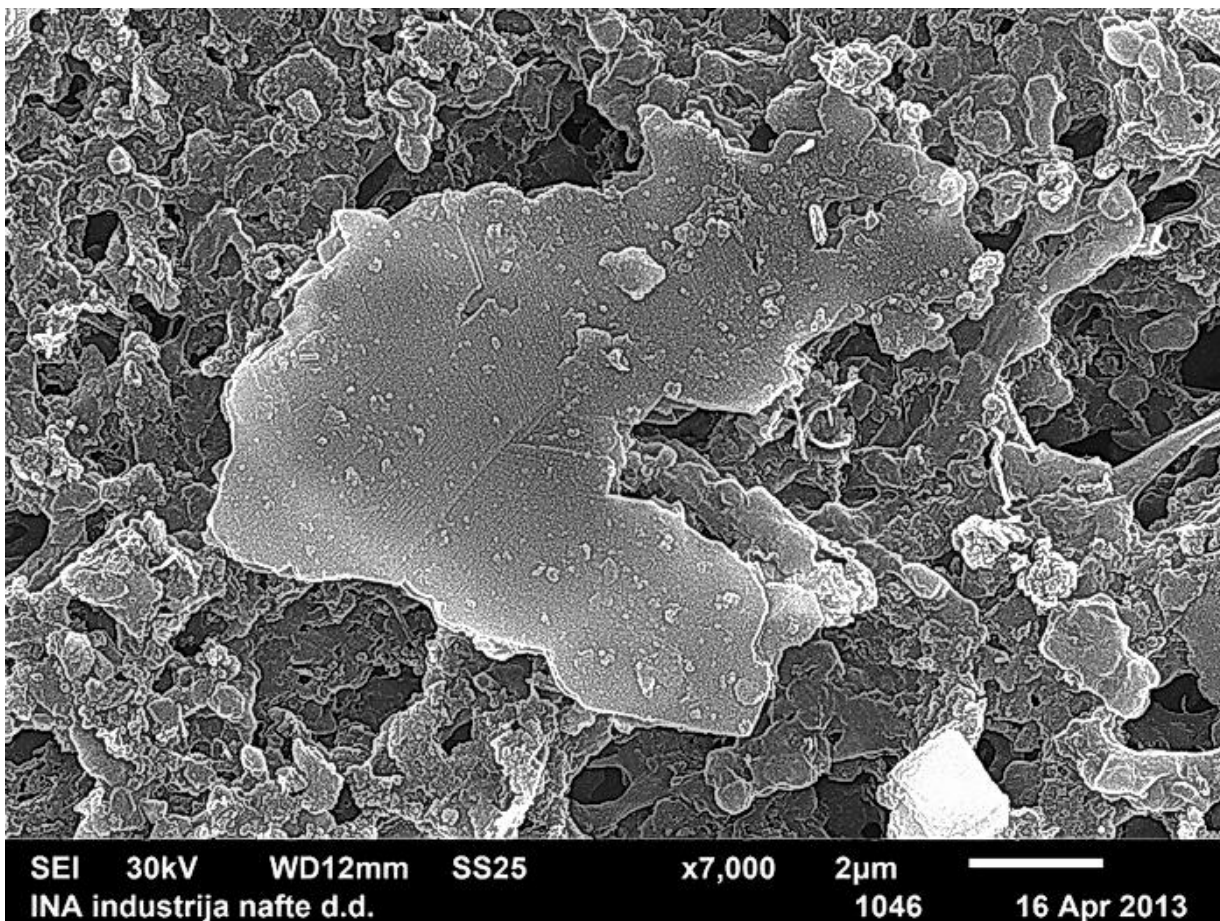
SAMPLE "0"

Photo 6. Sample „0“ micro photo, increasing 7.000x

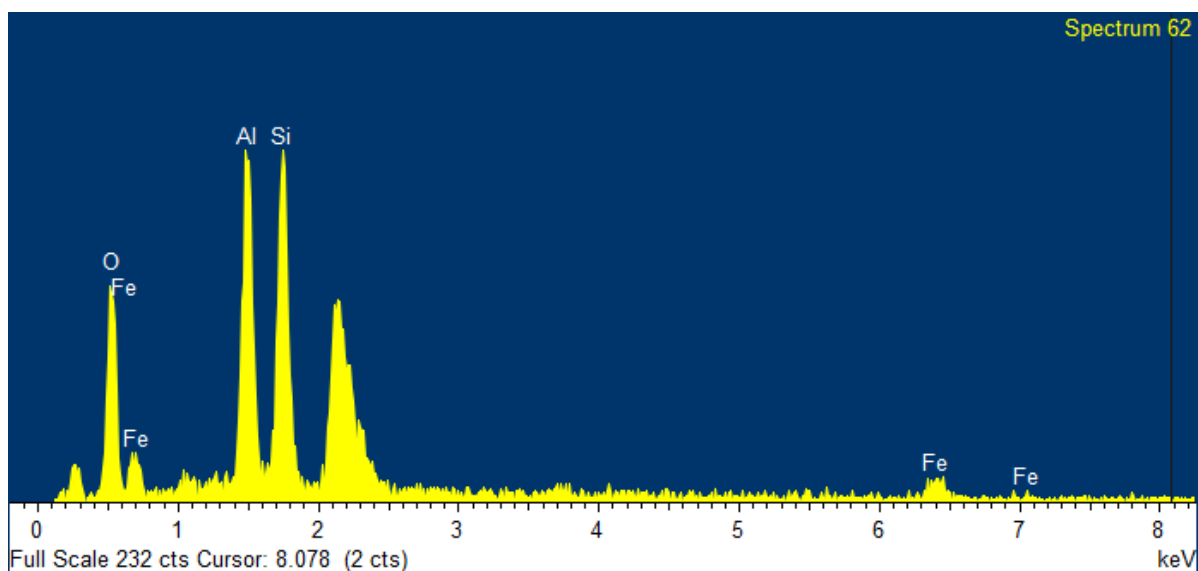
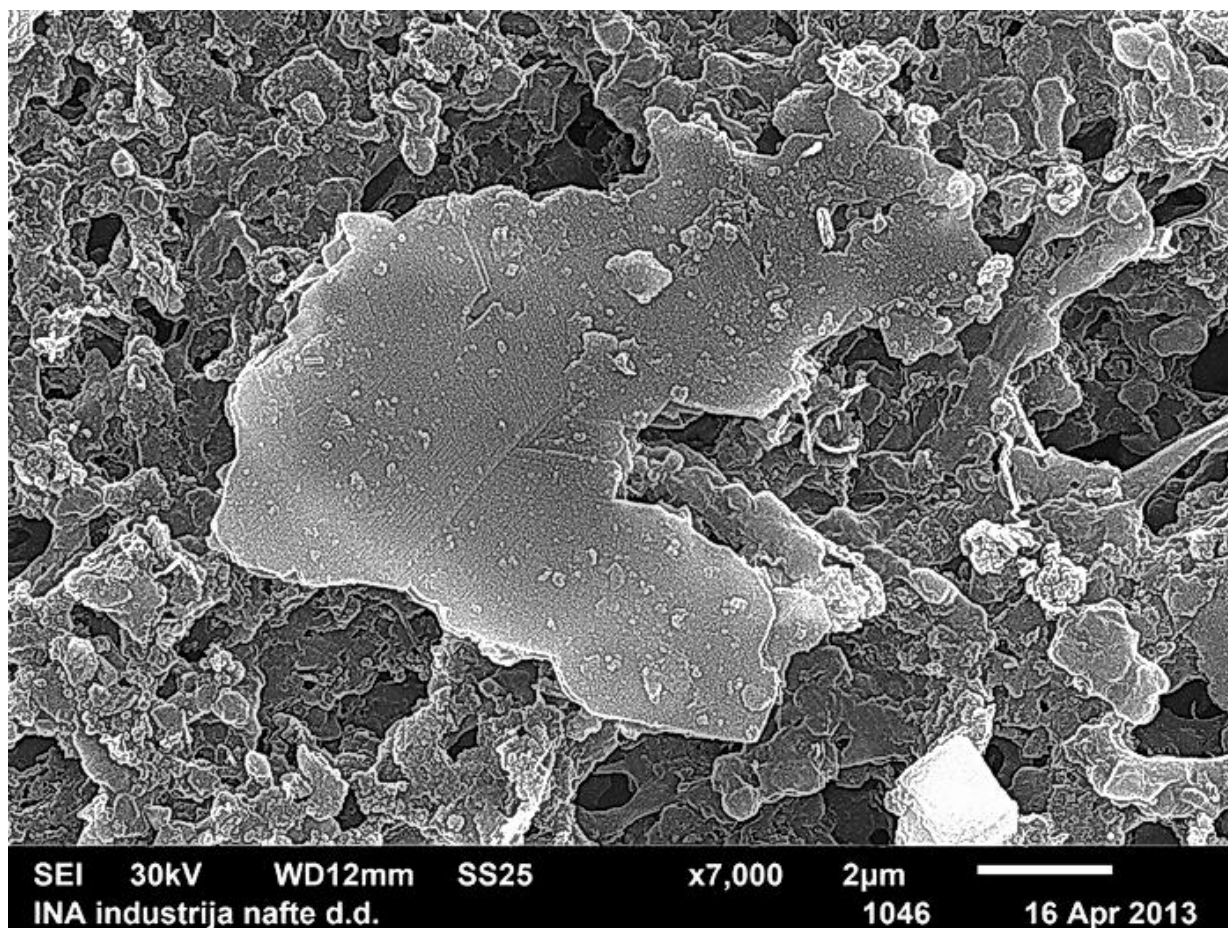


Photo 6a. EDX analysis of particles from photos 6 to 10

Table 2. Quantity chemical analysis of photo 6a

Element	Weight%	Atomic%	Spectrum 62
O K	64.16	76.40	
Al K	15.25	10.77	
Si K	17.22	11.68	
Fe K	3.37	1.15	
Totals	100.00		

**Photo 7. Sample „0“ micro photo, increasing 10.000x**

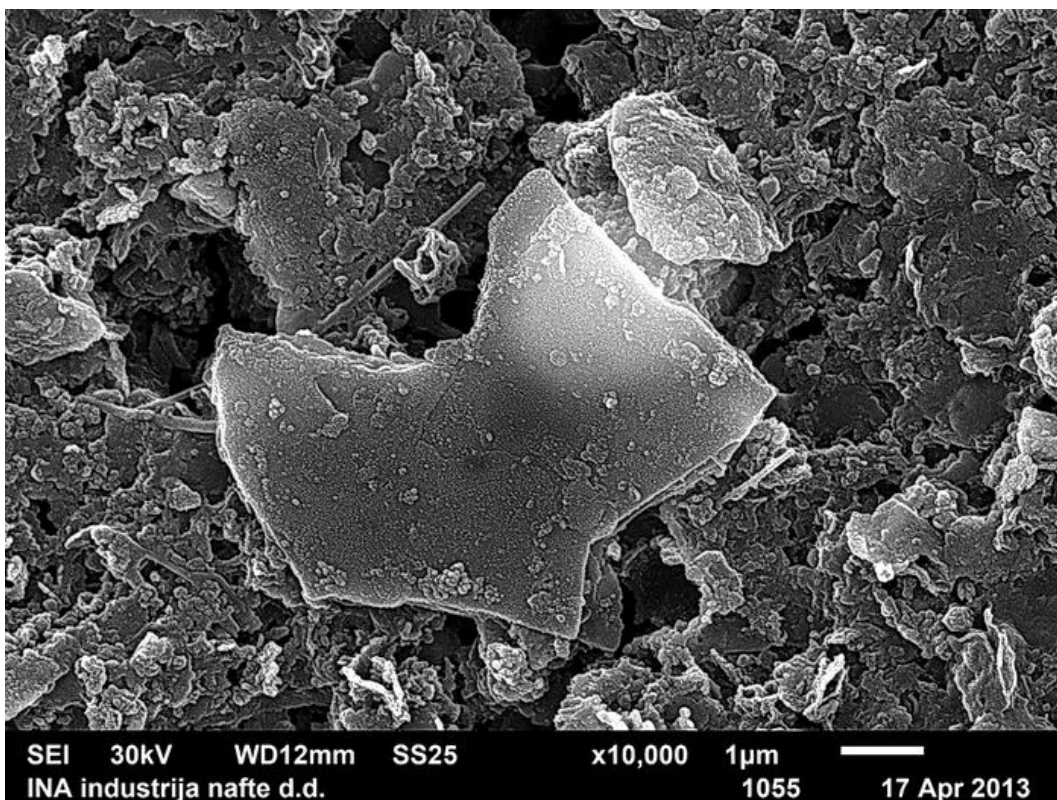


Photo 8. Sample „0“ micro photo, increasing 10.000x

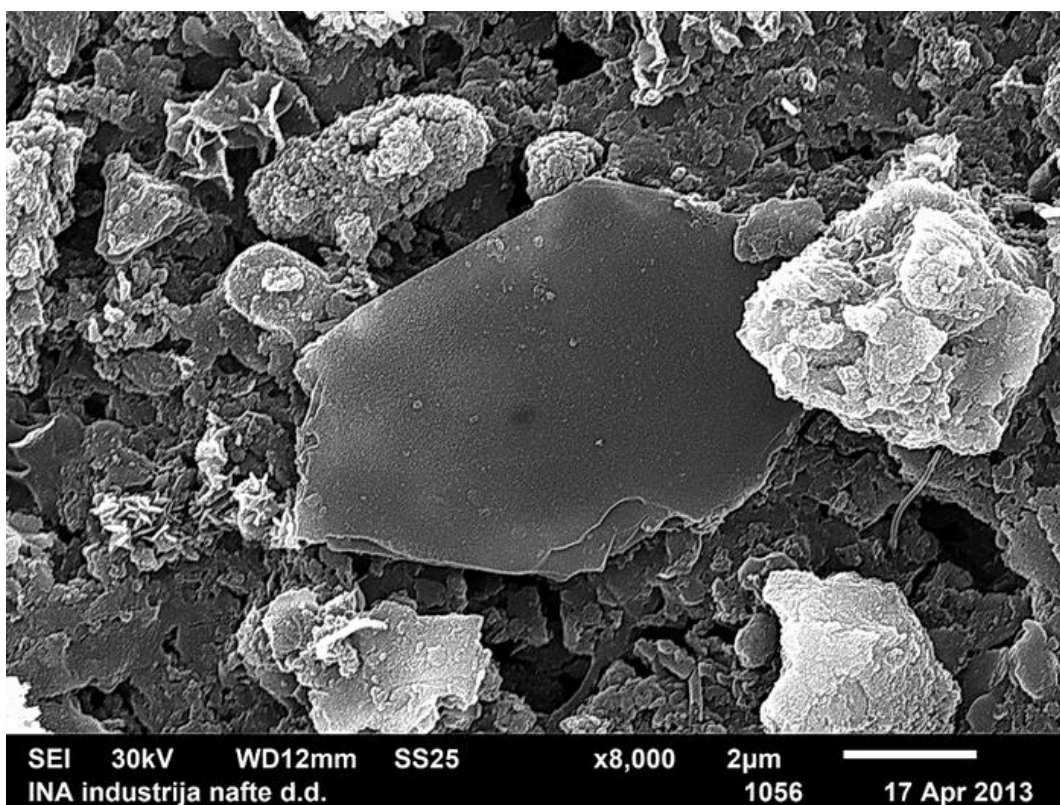


Photo 9. Sample „0“, micro photo, increasing 8.000x

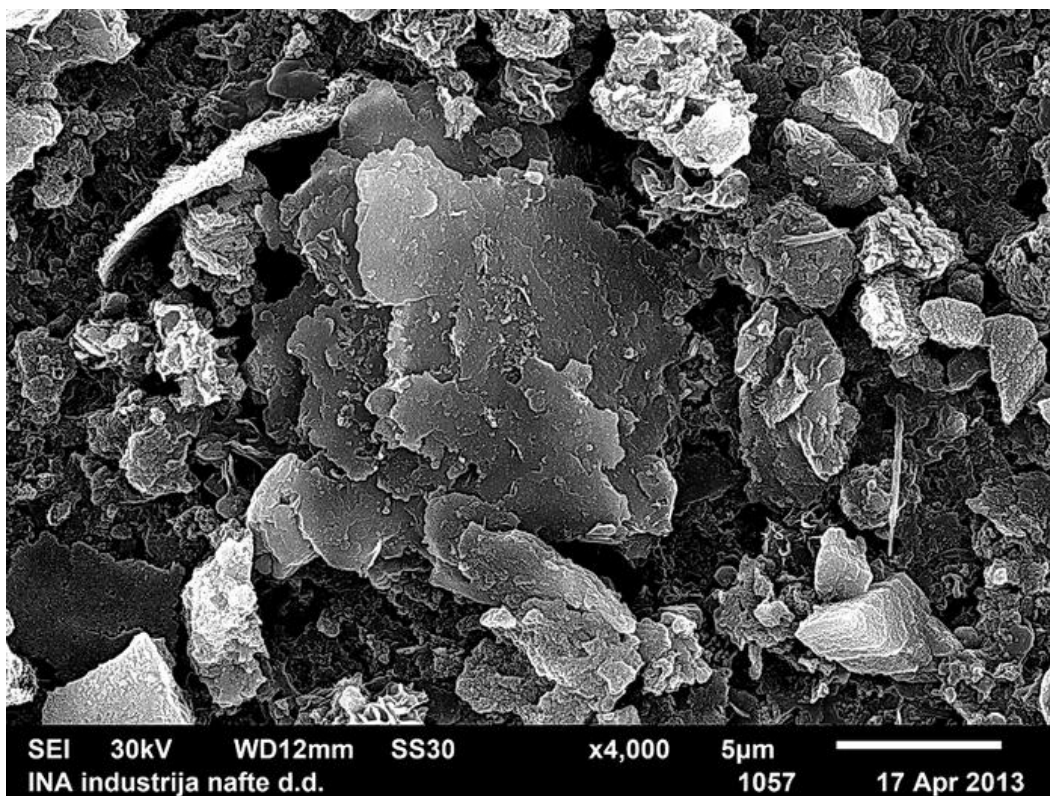


Photo 10. Sample „0“, micro photo, increasing 4.000x

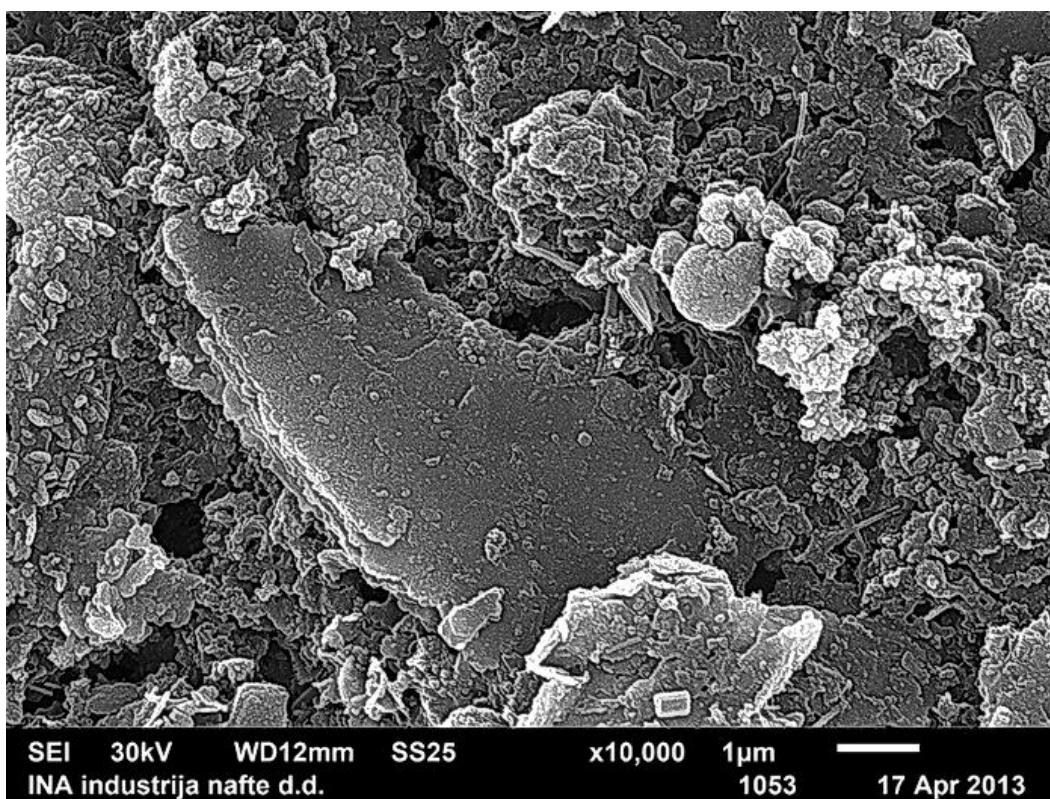


Photo 11. Sample „0“ micro photo, increasing 10.000x

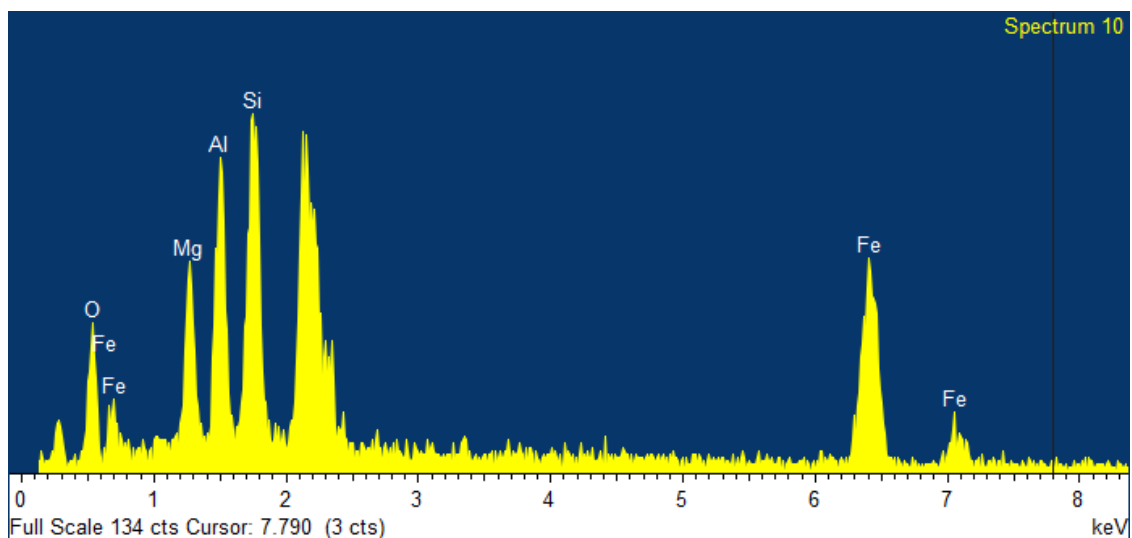


Photo 11a.

EDX particles on the photo 11

Table 3. Quantity chemical analysis from the photo 11a

Element	Weight%	Atomic%	Spectrum 10
O K	34.29	52.79	
Mg K	9.46	9.58	
Al K	12.66	11.55	
Si K	15.72	13.78	
Fe K	27.87	12.29	
Totals	100.00		

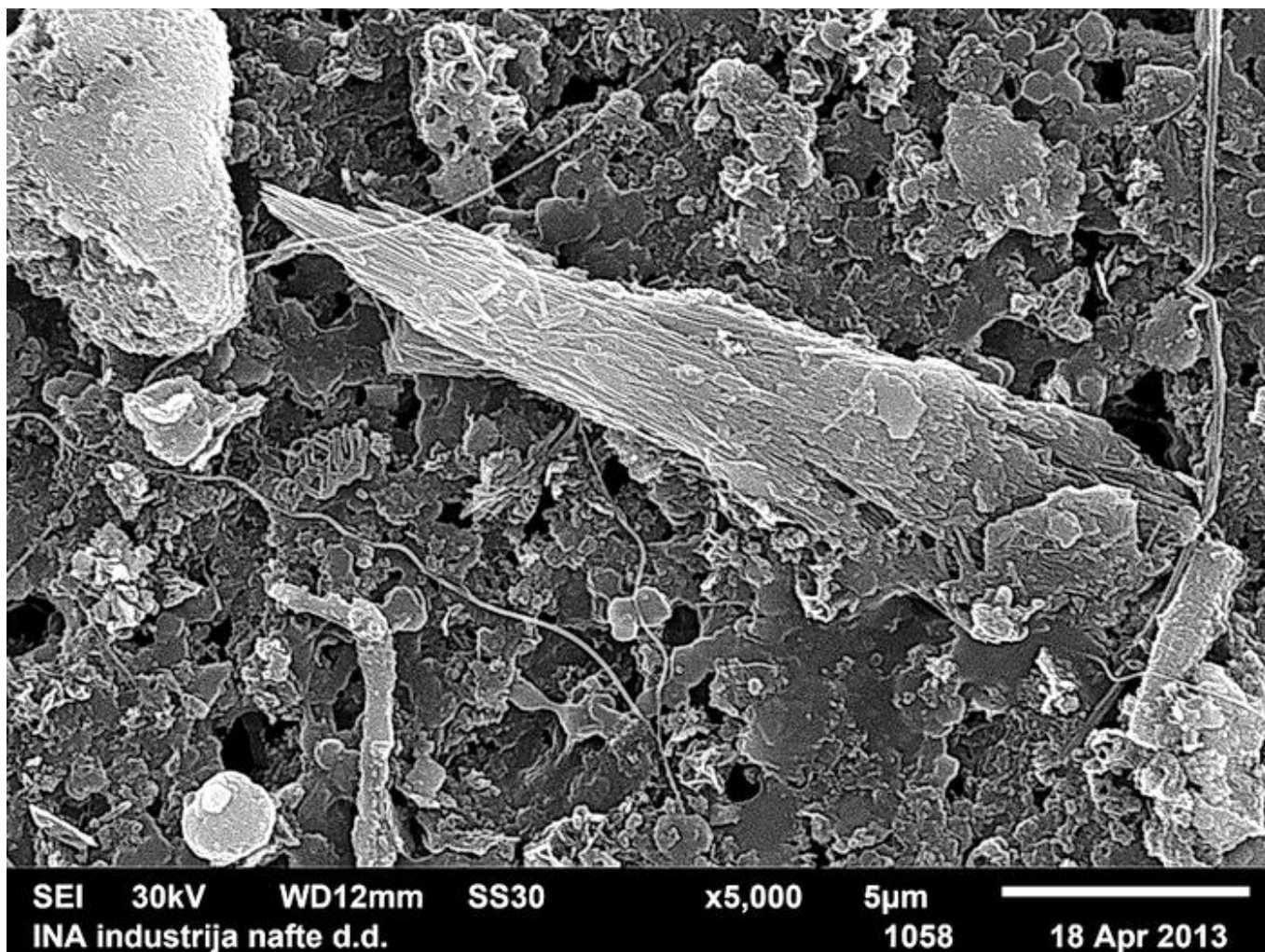


Photo 12. Sample „0“ micro photo, increasing 5.000x

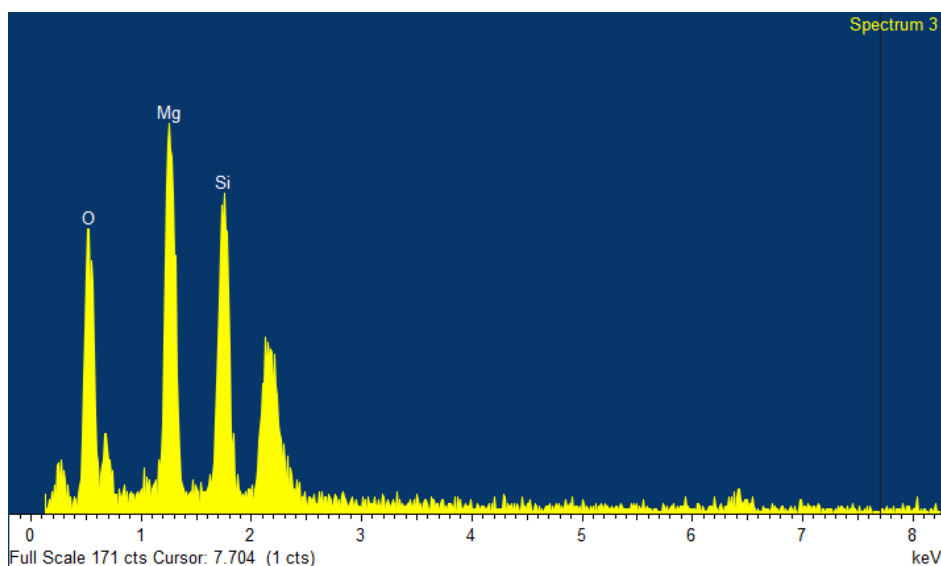
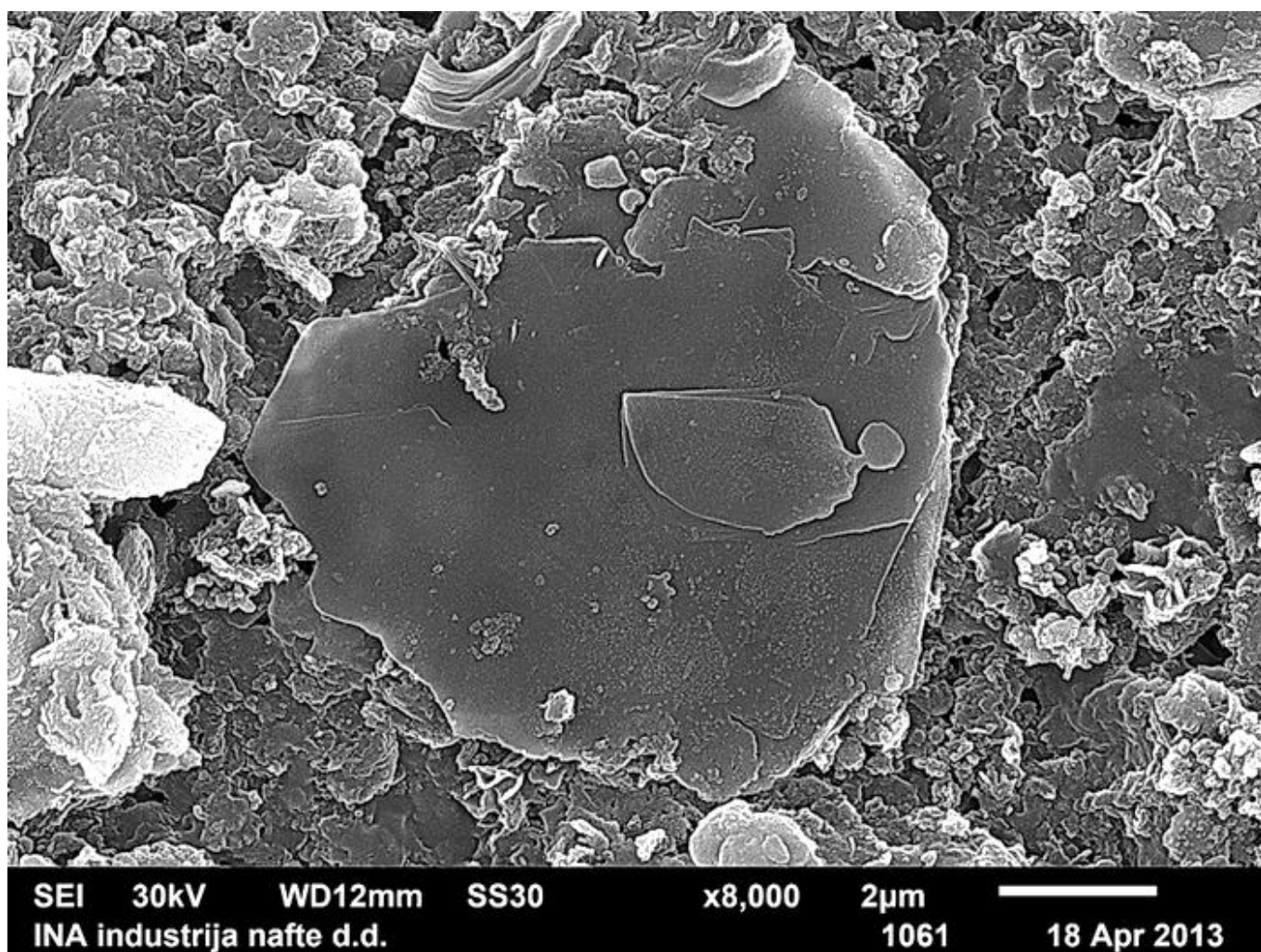


Photo 12a. EDX analysis of fibre particles from the photo 12

Table 4. Quantity chemical analysis from the photo 12a

Element	Weight%	Atomic%	Spectrum 3
O K	66.11	75.83	
Mg K	20.01	15.10	
Si K	13.88	9.07	
Totals	100.00		

SAMPLE "2"**Photo 13. Sample „2“ micro photo, increasing 8.000x**

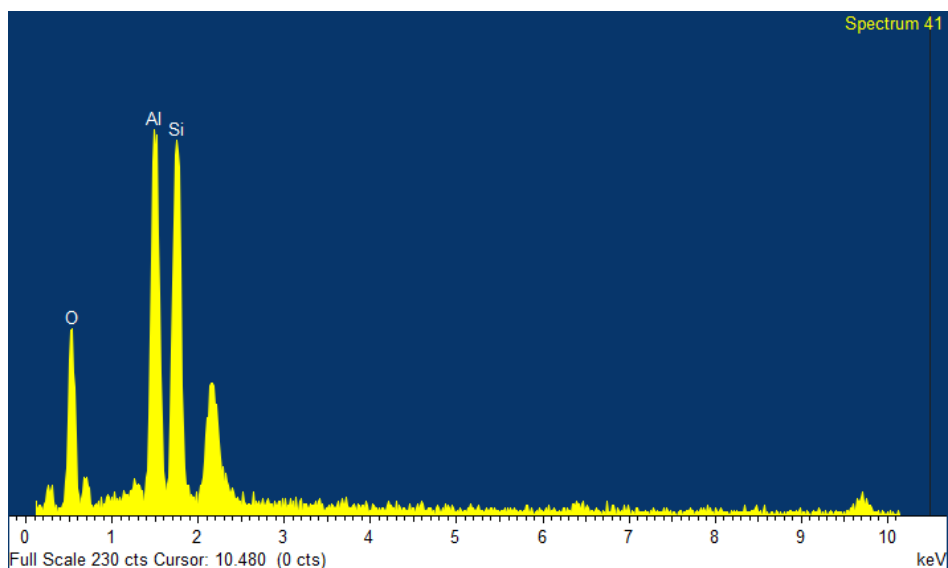


Photo 13a. EDX particles from the photos no. 13 to 17

Table 5. Quantity chemical analysis from the photo no. 13a

Element	Weight%	Atomic%	Spectrum 41
O K	62.39	74.08	
Al K	17.56	12.36	
Si K	20.05	13.56	
Totals	100.00		

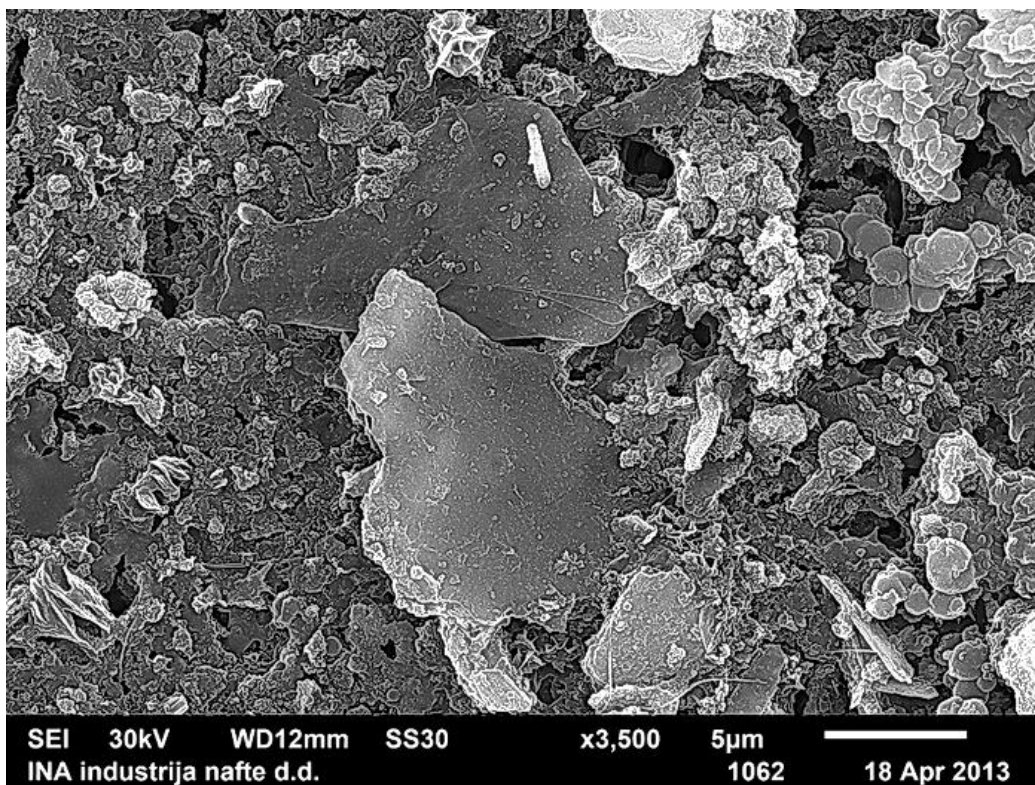


Photo 14. Sample „2“ micro photo, increasing 3.500x

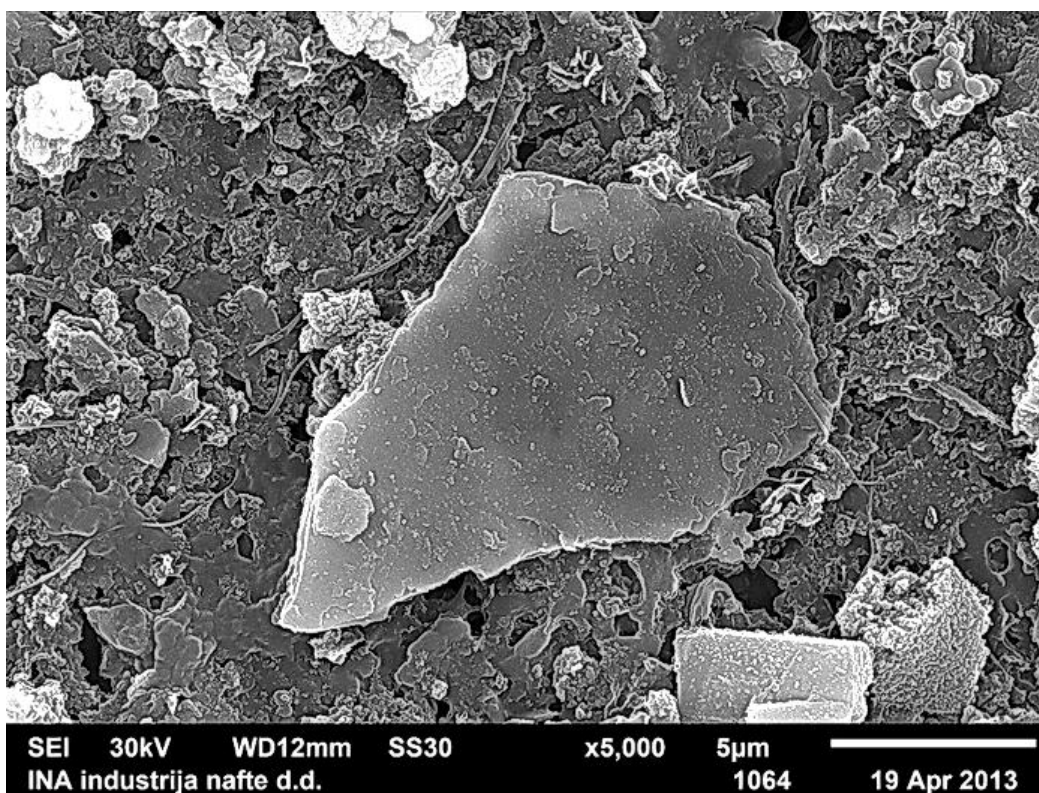


Photo 15. Sample „2“ micro photo, increasing 5.000x

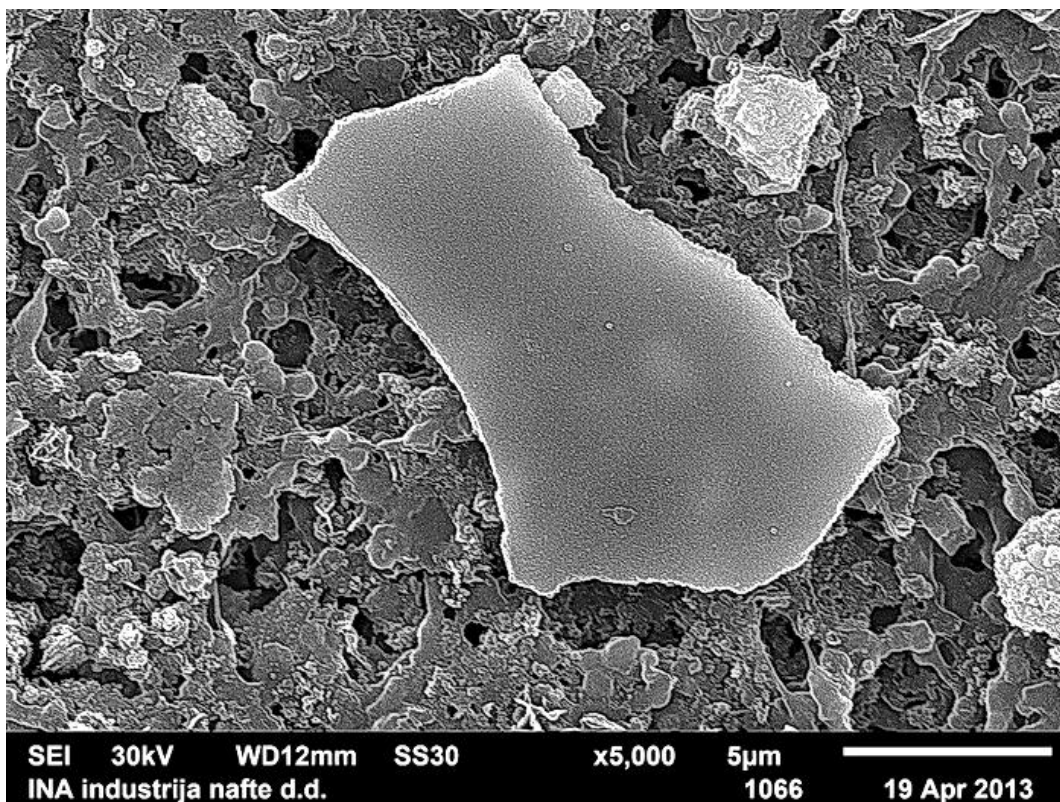


Photo 16. Sample „2“ micro photo, increasing 5.000x

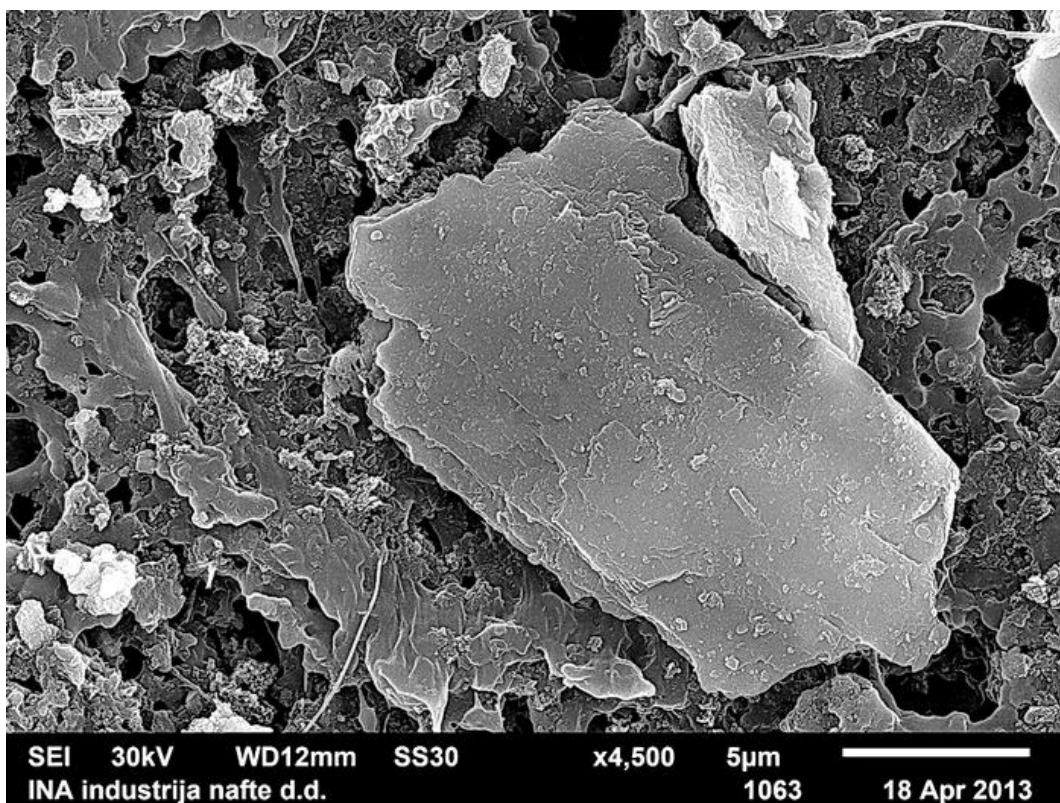


Photo 17. Sample „2“ micro photo, increasing 4.500x

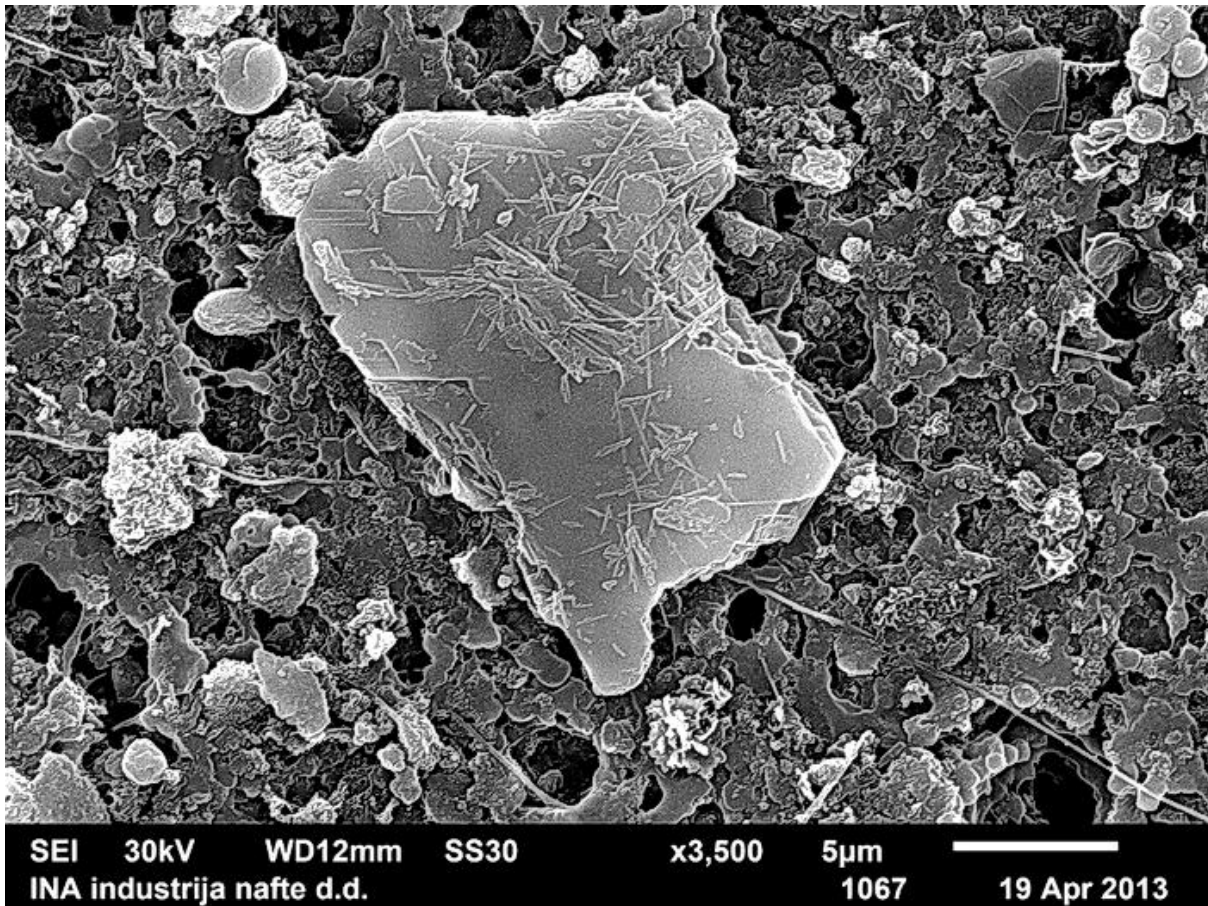


Photo 18. Sample „2“ micro photo, increasing 3.500x

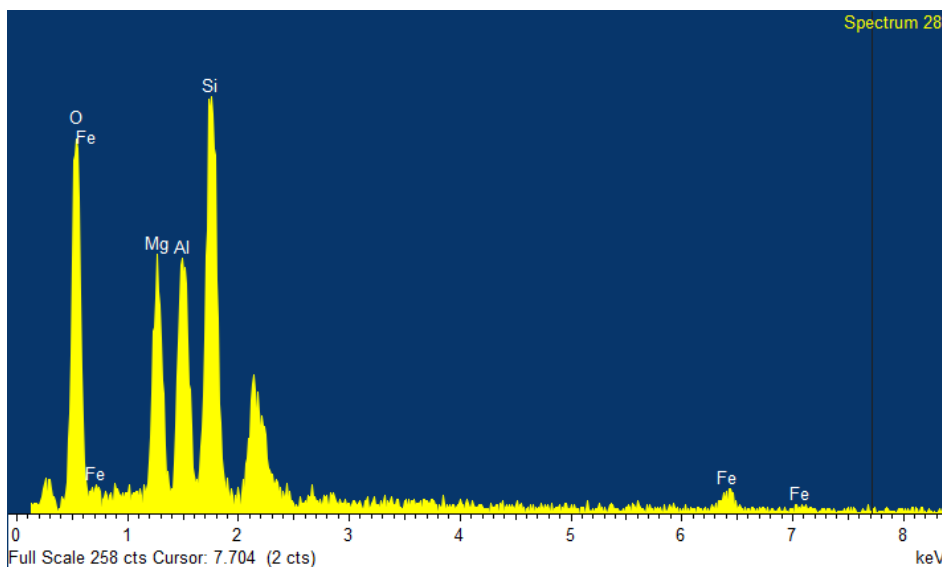
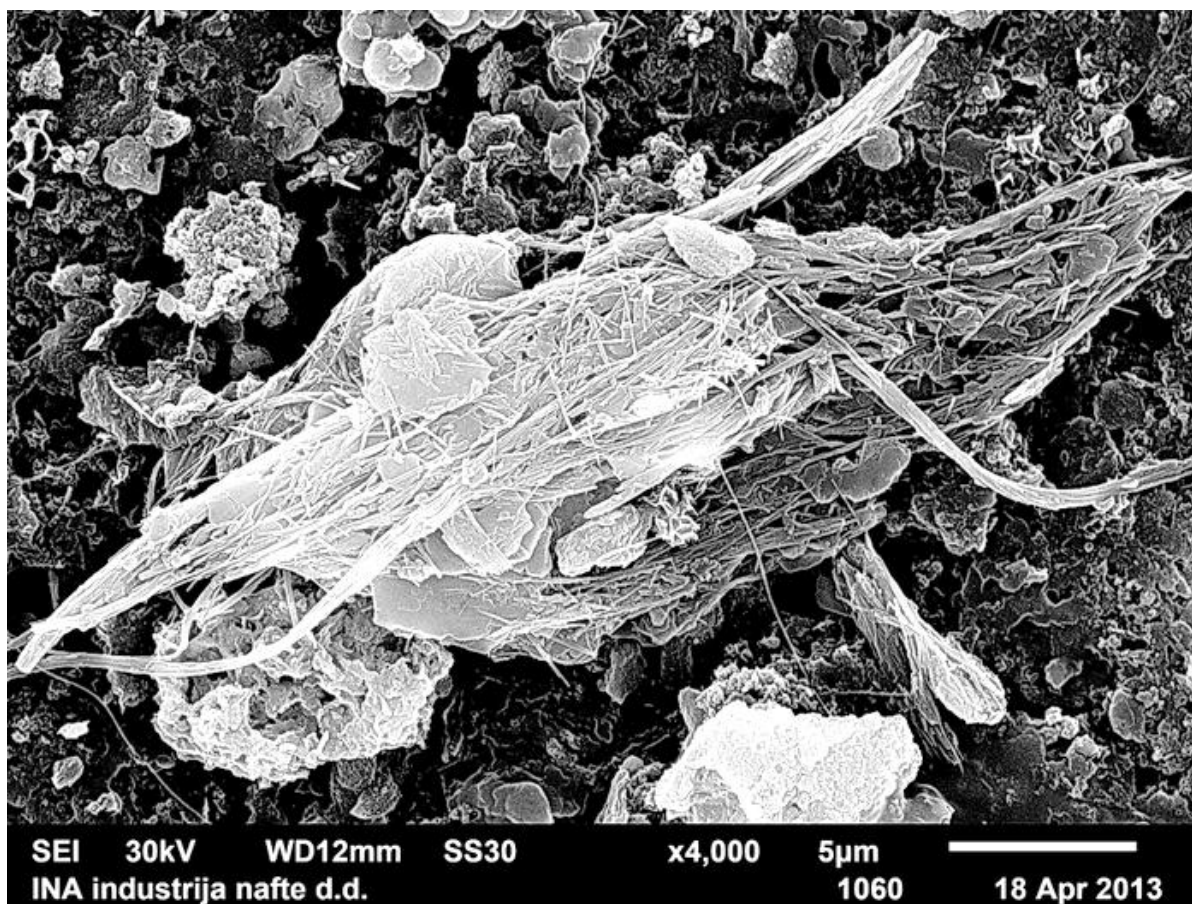


Photo 18a. EDX particles from photo no. 18

Table 6. quantity chemical analysis from the photo 18a

Element	Weight%	Atomic%	Spectrum 28
O K	66.69	77.45	
Mg K	9.14	6.99	
Al K	8.09	5.57	
Si K	14.14	9.35	
Fe K	1.93	0.64	
Totals	100.00		

**Photo 19. Sample „2“ micro photo, increasing 4.000x**

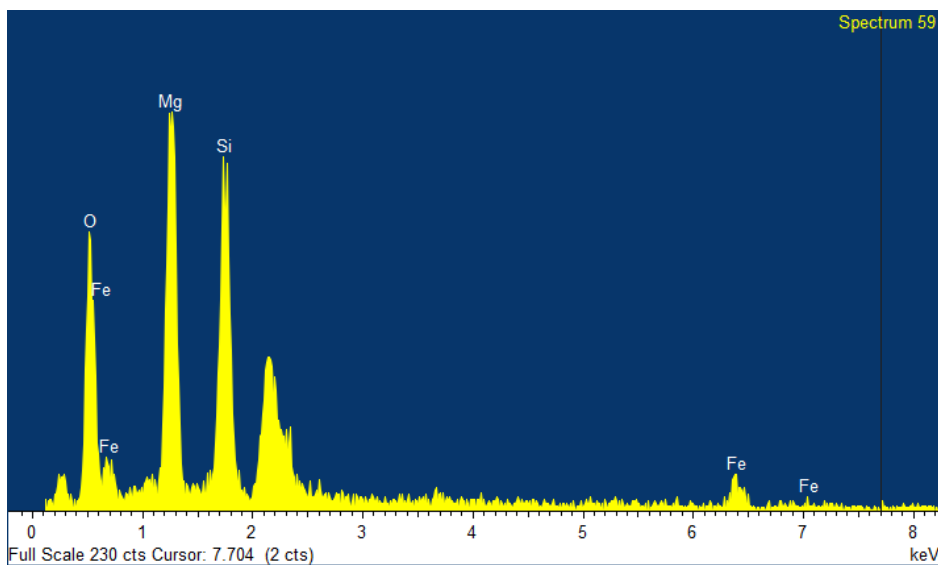


Photo 19a. EDX particles from the photo 19

Table 7. Quantity chemical analysis from the photo 19a

Element	Weight%	Atomic%	Spectrum 59
O K	61.68	73.12	
Mg K	20.57	16.05	
Si K	14.30	9.65	
Fe K	3.46	1.17	
Totals	100.00		

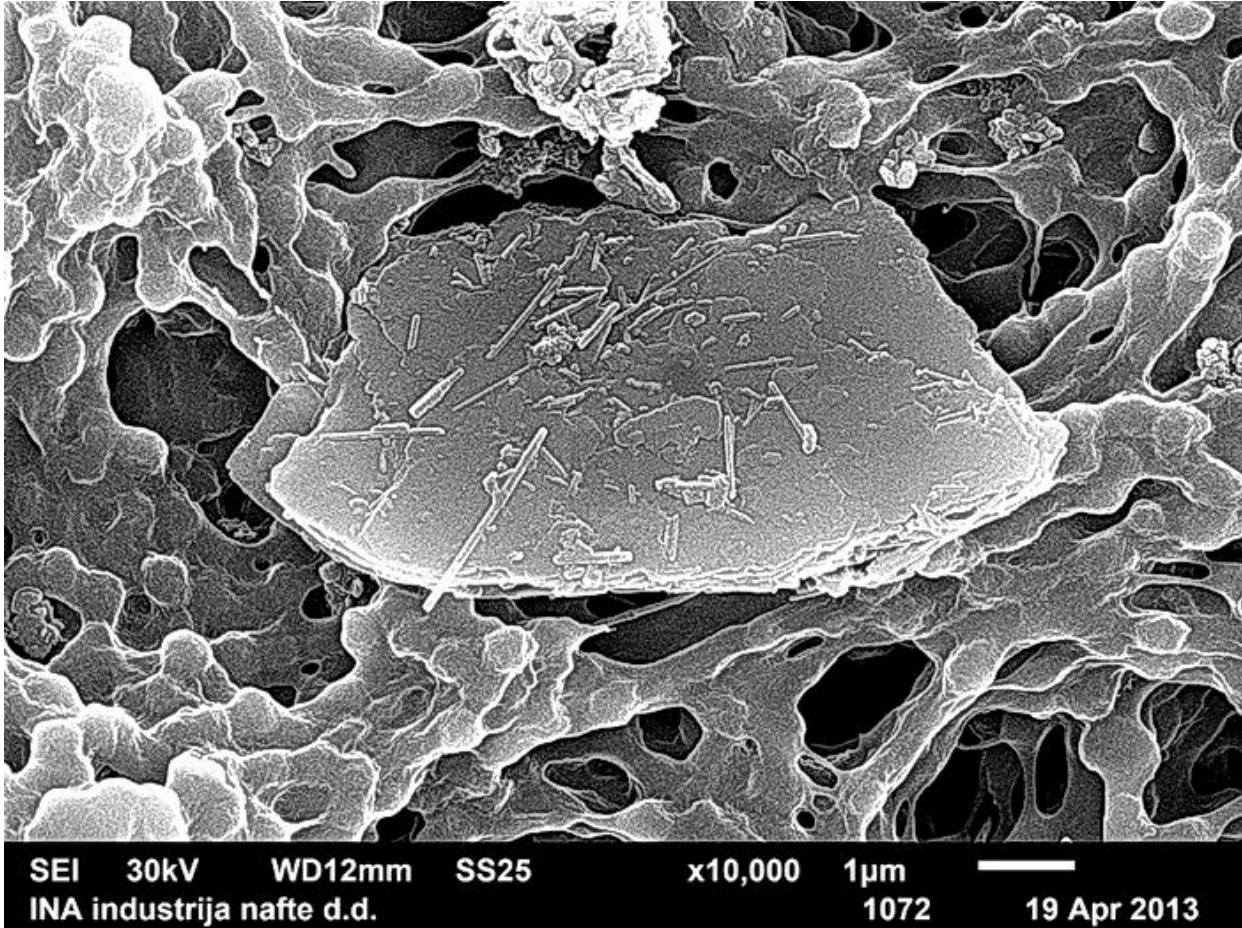
SAMPLE "5"

Photo 20. Sample „5“ micro photo, increasing 10.000x

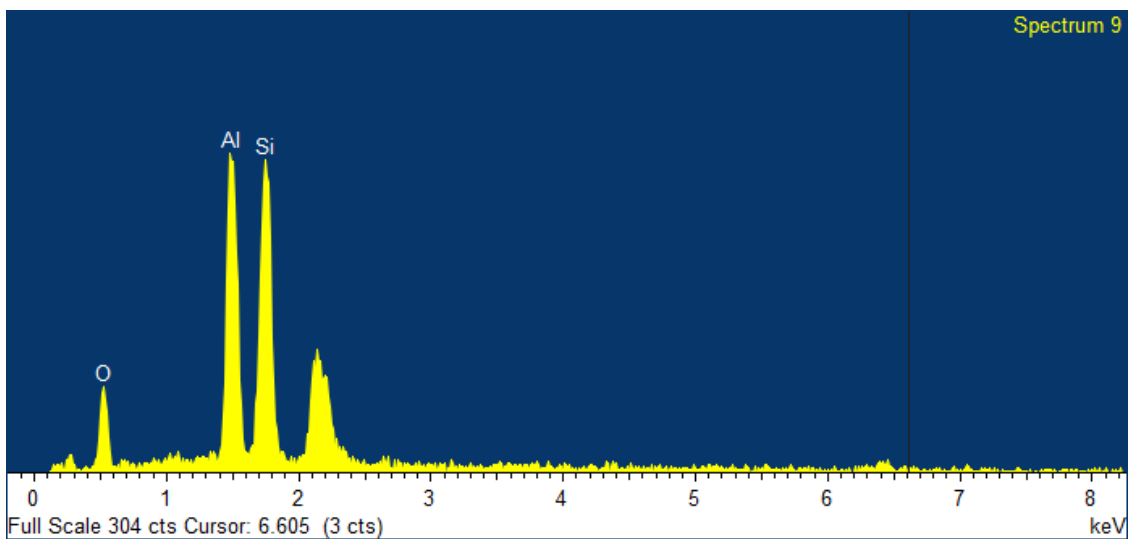
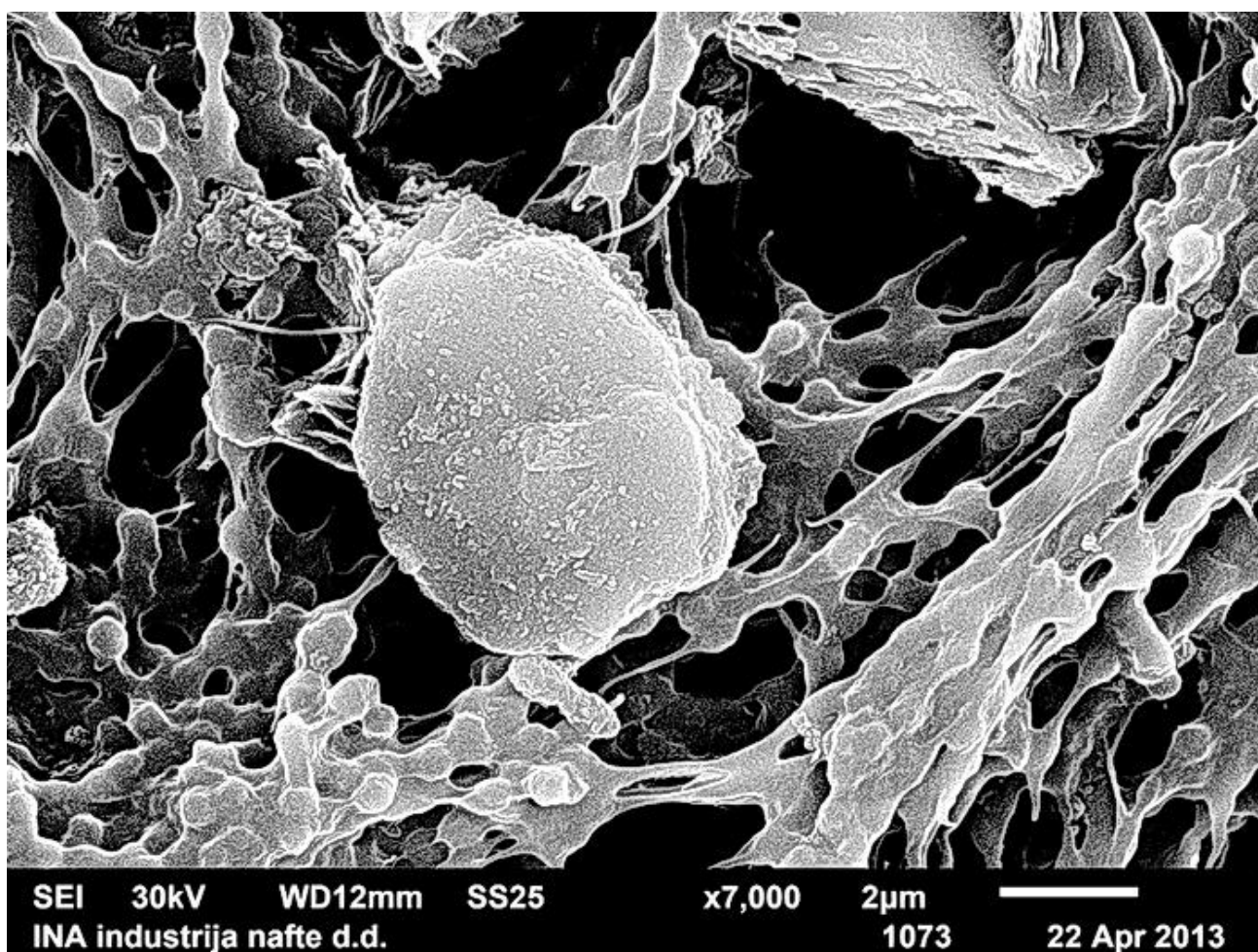


Photo 20a. EDX particles from the photos 20 to 23

Table 8. Quantity chemical analysis of the photo 20a

Element	Weight%	Atomic%	Spectrum 9
O K	50.49	63.75	
Al K	21.99	16.46	
Si K	27.52	19.79	
Totals	100.00		

**Photo 21. Sample „5“ micro photo, increasing 7.000x**

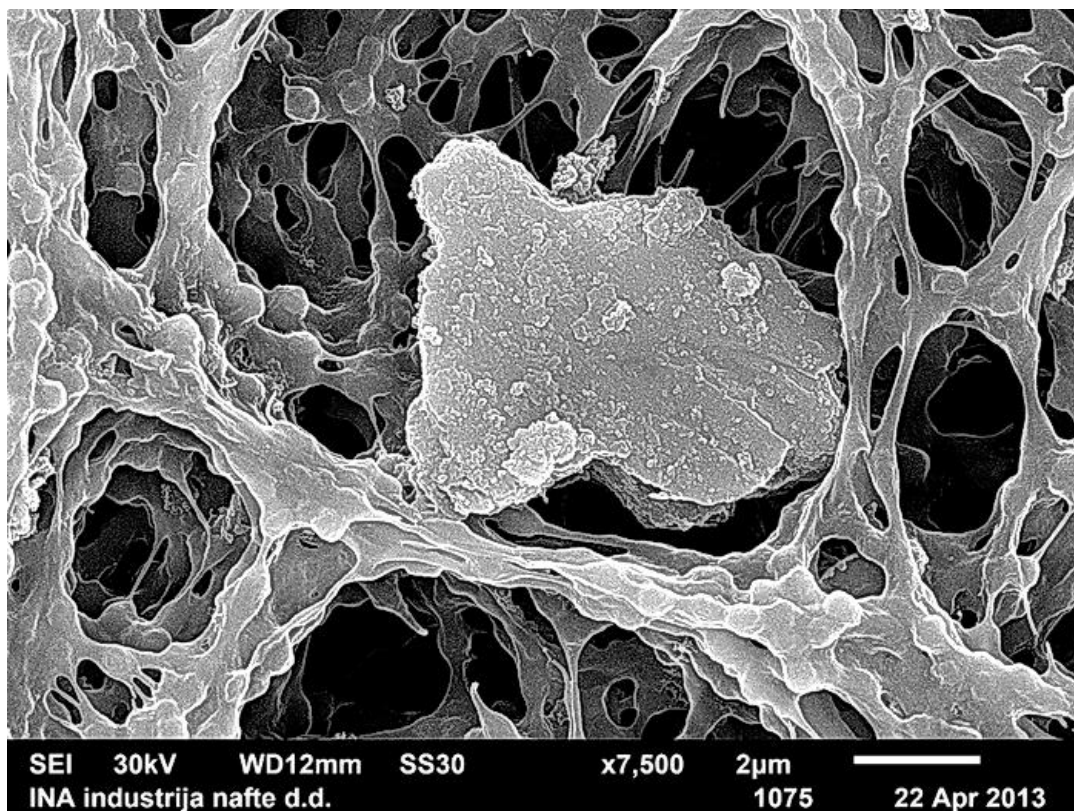


Photo 22. Sample „5“ micro photo, increasing 7.500x

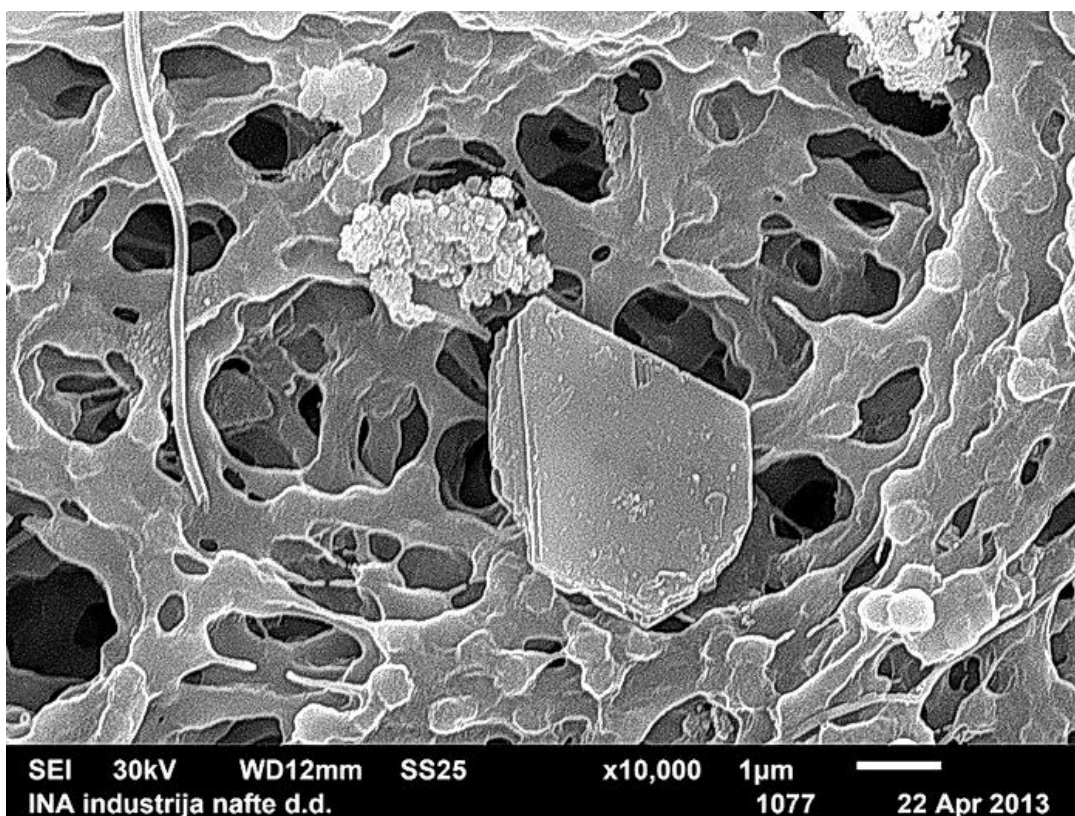
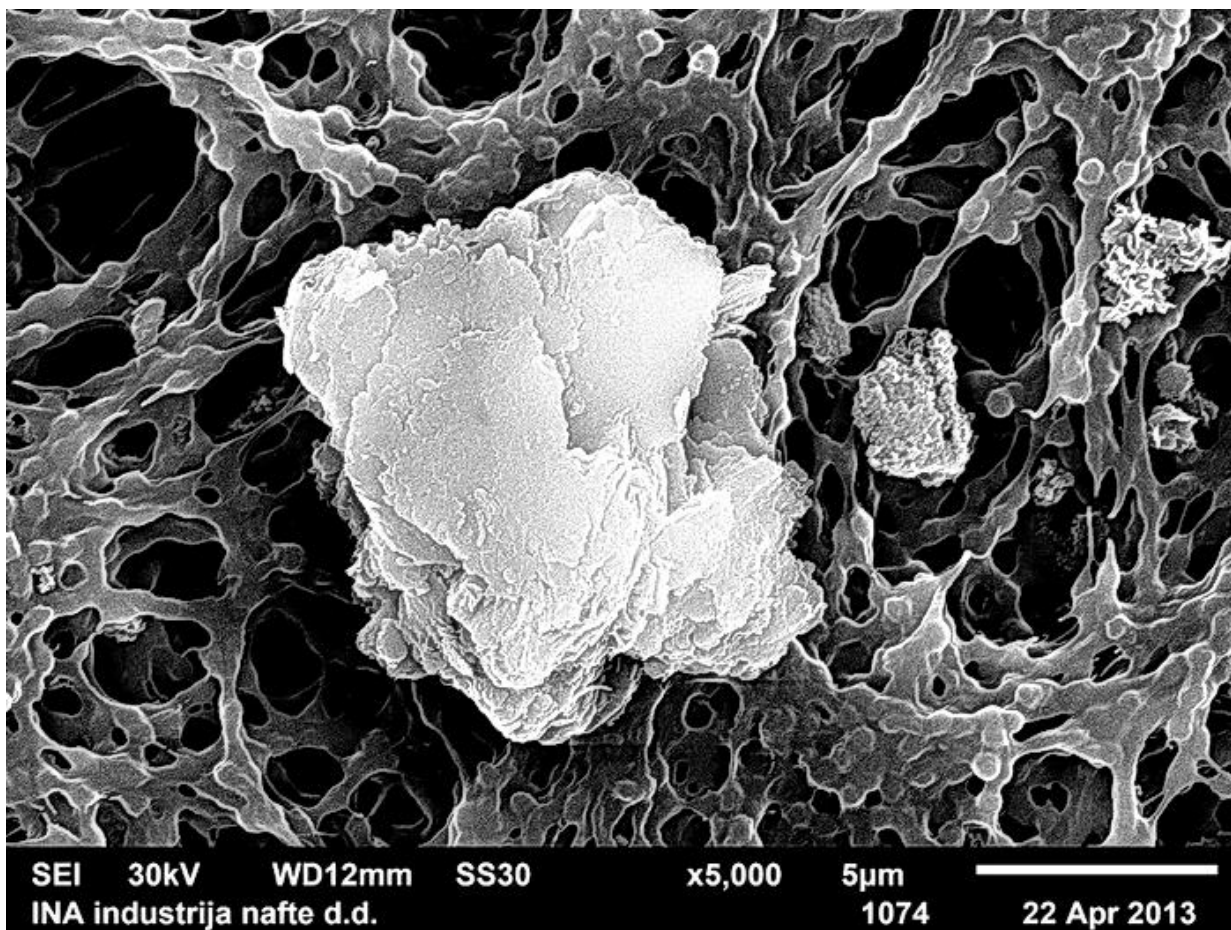
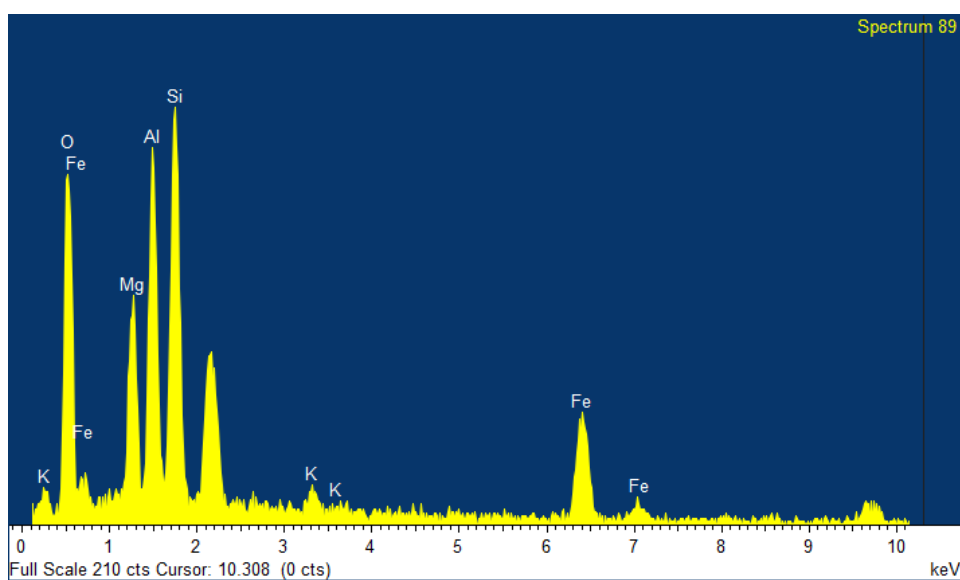


Photo 23. Sample „5“ micro photo, increasing 10.000x

**Photo 24. Sample „5“ micro photo, increasing 5.000x****Photo 24a. EDX particles from the photo 24**



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Table 9. Quantity chemical analysis from the photo 24a

Element	Weight%	Atomic%	Spectrum 89
O K	60.00	74.19	
Mg K	7.49	6.09	
Al K	10.20	7.48	
Si K	12.04	8.48	
K K	0.78	0.40	
Fe K	9.49	3.36	
Totals	100.00		

-----KRAJ IZVJEŠTAJA-----